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Indian Standard

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(Reaffirmed 2021)

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(दूसरा पुनरीक्षण)

Acetoacetic Methyl Ester — Specification

(Second Revision)

ICS 71.080.70

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Price Group 2

Organic Chemicals, Alcohols and Allied Products and Dye Intermediates Sectional Committee, PCD 9

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Organic Chemicals, Alcohols and Allied Products and Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Acetoacetic methyl ester ($C_5H_8O_3$) is an important organic intermediate used in the used in the manufacture of dyestuffs. It is represented by the following chemical formula:

$CH_3.CO.CH_2.CO.O.CH_3$
ACETOACETIC METHYL ESTER
(Molecular Mass 116)

This standard was first published in 1978 stipulating the requirements and methods of test for Acetoacetic methyl ester. The first revision of this standard was brought in 1987 to modify the requirements for Acetoacetic methyl ester content from 99.0 percent to 98.5 percent*, Min,* acidity from 0.3 percent to 0.2 percent, *Max*, and moisture content from 0.2 percent to 0.1 percent, *Max*. In addition to the existing hydroxylamine hydrochloride method, gas chromatographic (GC) method of test for Acetoacetic methyl ester introduced.

In this revision the requirements for Acetoacetic methyl ester content modified from 98.5 percent to 99.0 percent, *Min* and for acidity from 0.2 percent to 0.1 percent, *Max*, respectively. The gas chromatographic (GC) method of test for Acetoacetic methyl ester also upgraded.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

ACETOACETIC METHYL ESTER — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Acetoacetic methyl ester.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibilities of applying the most recent editions of the standards indicated below.

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless liquid.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in this standard.

Table 1 Requirements for Acetoacetic Methyl Ester
(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl. No.	Characteristic	Requirement	Method of Test/Ref to Annex
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	99.0	A
ii)	Acidity (as CH_3COOH), percent by mass, *Max*	0.1	B
iii)	Moisture, percent by mass, *Max*	0.1	C

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) or as agreed to between the purchaser and the supplier. The container shall be securely closed.

4.2 Marking

4.2.1 Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number or lot number, in code or otherwise;

d) Date of packing; and

e) Tare, net and gross mass.

4.2.2 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

4.2.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS 5299.

5.2 Number of Tests

5.2.1 Each individual sample shall be tested for assay.

5.2.2 Tests for remaining characteristics, namely, acidity and moisture shall be conducted on the composite sample.

5.3 CRITERIA FOR CONFORMITY

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of assay, if each of individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the

requirements of all other characteristics tested on the composite sample given in **5.2.2**, the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out as prescribed in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF ACETOACETIC METHYL ESTER

A-1 ASSAY

A-1.1 General

Two methods have been prescribed for assay: (a) hydroxylamine hydrochloride method and (b) gas chromatographic method. In case of dispute, gas chromatographic method shall be used as the referee method.

A-1.2 Hydroxylamine Hydrochloride Method

A-1.2.1 *Outline of the Method*

Acetoacetic methyl ester reacts with hydroxylamine hydrochloride and librates hydrochloric acid which is estimated.

A-1.2.2 *Reagents*

A-1.2.2.1 *Methanol*

A-1.2.2.2 *Hydroxylamine hydrochloride solution* — approximately 1N.

Weigh about 35 g of pure hydroxylamine hydrochloride in a beaker, dissolve it in about 100 ml of water and make it up to 500 ml in a standard volumetric flask with water.

A-1.2.2.3 *Sodium hydroxide solution* – 1 N

A-1.2.2.4 *Methyl orange indicator*

A-1.2.2.5 *Methyl blue indicator*

A-1.2.3 *Procedure*

Weigh accurately about 2.5 to 3 g of sample in a 250-ml stoppered conical flask. Add to this 50 ml of methanol followed by 50 ml of hydroxylamine hydrochloride solution. Mix well and allow to stand for one to one and half hours at room temperature with occasional shaking. Add 5 drops of methyl orange indicator and 5 drops of methylene blue indicator and titrate against sodium hydroxide solution till colour change is violet to light green. Take a blank in the similar way.

A-1.2.4 *Calculation*

$$\text{Assay, percent by mass} = \frac{(V_1 - V_2) \times 11.6 \times N}{M}$$

where

V_1 = volume in ml of sodium hydroxide solution required for the sample,

V_2 = volume in ml of sodium hydroxide solution required for the blank,

N = normality of sodium hydroxide solution, and

M = mass in g of sample taken for the test.

A-1.3 Gas Chromatographic Method

A-1.3.1 *Outline of the Method*

The assay of Acetoacetic methyl ester is determined by gas liquid chromatographic method. A sample of the material is injected into the gas chromatograph apparatus where it is carried by the carrier gas from one end to other end. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after the other and are detected by suitable means where response is related to the amount of specific component leaving the column. The chromatographic conditions given here are for guidance only. The typical chromatogram for Acetoacetic methyl ester is shown in Fig.1.

A-1.3.2 *Apparatus*

A-1.3.2.1 *Gas chromatograph*

Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used.

A-1.3.2.2 *Column Details*

Column	BP-10
Length	25 meter
Diameter	0.53 mm
Film thickness	1 µm

A-1.3.2.3 *Detector type:* Flame Ionization Detector (FID)

A-1.3.3 *Operating Parameters of Gas Chromatograph*

a) Oven Temperature : 50°C for 2 min and 109°C for 10.6 min at the rate of 8°C per minute

b) Injector Temperature : 200°C

c) Detector Temperature : 200°C

d) Quantity Injected : 0.4µl (neat)

e) Carrier Gas (N_2) : 5 ml/min

f) Attenuation : 32

g) Calculation : Area normalization with response factors

h) Run Time : 20 min

j) Retention Time : Methanol = 1.52 min; Acetone = 1.84 min Acetoacetic methyl ester (AAME) = 8.20 min

k) Response Factor : Methanol = 1.00; Acetone = 0.6 and Acetoacetic methyl ester (AAME) = 1.00

A-1.3.4 *Procedure*

Conduct the flow of the carrier gas and inject 0.4 microlitre of the sample at the injection port where it is vapourized and well mixed with carrier gas. This led into the chromatographic column wherein the vapourized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For an efficient separation, the column should be maintained at the temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives a signal corresponding to the amount of particular constituent leaving the column. The detector signal on transmission to the recorder plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

A-1.3.5 *Calculation*

Calculate the peak areas of individual constituent pertaining to acetoacetic methyl ester as also the other constituents and calculate the purity of the sample as given below:

$$\text{Assay, percent} = \frac{A_1}{A_1 + A_2 + A_3 + \ldots\ldots + A_n} \times 100$$

where

A_1 = area under acetoacetic methyl ester peak;

A_2 = area under other peaks, say peak 1;

A_3 = area under other peaks, say peak 2, etc, and

n = number of other peaks apart from acetoacetic methyl ester.

FIG. 1 CHROMATOGRAM OF ACETOACETIC METHYL ESTER

ANNEX B

[*Table* 1, *Sl* No.(ii)]

DETERMINATION OF ACIDITY

B-1 REAGENTS

B-1.1 Mixed Indicator — Dissolve 0.05 g of bromocresol green and 0.1 g of methyl red in ethanol and make up to 100 ml.

B-1.2 Sodium Hydroxide Solution — 0.1 N.

B-2 PROCEDURE

Weigh accurately about 5 to 6 g of the material in a 250 ml conical flask and add about 100 ml of water and shake it till a good dissolution is obtained. Add a few drops of mixed indicator till the colour changes to red. Titrate the solution with sodium hydroxide solution till colour changes from red to green.

B-3 CALCULATION

$$\text{Acidity (as } CH_3COOH\text{), percent by mass} = \frac{V \times 0.6}{M}$$

where

V = volume in ml of sodium hydroxide used for titration, and

M = mass in g of the sample taken.

ANNEX C

[*Table* 1, *Sl* No. (iii)]

DETERMINATION OF MOISTURE

C-1 Determine the moisture content of the material as prescribed in IS 5299.

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Amendments Issued Since Publication

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